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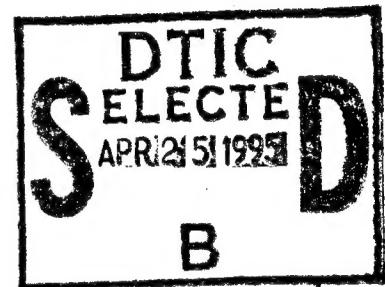
# **ZERO DISCHARGE ORGANIC COATINGS**

## **Powder Paint - UV Curable Paint - E-Coat**

Quarterly Technical Report  
January 1995 - March 1995

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### **Abstract**

The Zero Discharge Organic Coatings (ZDOC) R&D project will substantially advance coatings technology through the development of high performance zero-discharge coating systems. Development efforts are proceeding on three coating technologies, powder paint, Ultraviolet (UV) curable paint and electro-coating (E-coat) paint. These three paint technologies offer the potential of high performance coatings with no volatile organic compound (VOC) emissions or hazardous waste generation. These three technologies and their associated application processes will be applicable to a wide variety of military equipment and structures, thereby eliminating volatile organic compound (VOC's) and toxic heavy metals from coatings and painting operations.

The ZDOC team of Hughes Aircraft Company, Lehigh University, University of Arizona, and the Naval Air Warfare Center Aircraft Division Warminster offers a blend of experience, expertise, and capabilities in all aspects of organic coatings technology. This quarterly report reviews the team's technical activities and results during the period January 1995 - March 1995.

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# **ZERO DISCHARGE ORGANIC COATINGS**

## **Powder Paint - UV Curable Paint - E-Coat**

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### **List of Acronyms and Abbreviations**

CRADA	Cooperative Research and Development Agreement
E-Coat	Electrocoat
HAC	Hughes Aircraft Company
HMSC	Hughes Missile Systems Company
IR	Infra Red
NAWCADWAR	Naval Air Warfare Center - Aircraft Division - Warminster, Pa
UofA	University of Arizona
UV	Ultraviolet
ZDOC	Zero Discharge Organic Coatings
TPE	Total Performance Evaluation

# ZERO DISCHARGE ORGANIC COATINGS

## Powder Paint - UV Curable Paint - E-Coat

### SUMMARY

This report summarizes the research efforts of the ZDOC project team during the three month period from January 1995 through March 1995. The ZDOC project team is comprised of 5 teams from different organizations (HMSC, NAWCADWAR, HAC-TSD, Lehigh & UofA) each working in separate but related portions of the overall ZDOC project. The development of inhibited powder coatings has completed testing of the first iteration of coatings and the second iteration is presently being tested. Five UV curable inhibited paint systems are still under consideration after screening many candidate materials and testing on these systems is continuing. Testing to characterize and model non-toxic corrosion inhibitors is continuing and development of electro-coatings is continuing the establishment of an in-house E-coat process line. Team activities and accomplishments during this quarter include:

- Completed second iteration of inhibited powder coatings and testing was initiated.
- Continued testing of UV curable inhibited paint systems.
- Began evaluating the effects of antistats on electromagnetic signals when applied to non-conductive substrates.
- Began evaluating the effects of antistats on the barrier properties of epoxy coatings.
- Continued testing resin systems for identifying mechanism of corrosion inhibitors.
- Produced five pigment pastes containing individual corrosion inhibiting pigments for E-coat.
- 20 panels were E-coated using the five pigmented pastes.
- Began performance evaluation on E-coated panels containing inhibitors.

### 1.0 INTRODUCTION

Organic coatings, which provide the primary defense against environmental degradation of military equipment, have been identified as a major source of hazardous material emissions and waste in the Department of Defense. The current approach to solving this problem is through the incremental reduction of the coatings' toxic components. In contrast, the Zero Discharge Organic Coatings (ZDOC) R&D project will substantially advance coatings technology through the development of high performance zero-discharge coating systems. Development efforts are proceeding on three coating technologies: powder paint, ultraviolet (UV) curable paint and electro-coating paint. These three paint technologies offer the potential of high performance coatings with no volatile organic compound (VOC) emissions or hazardous waste generation. These three technologies and their associated application processes will be applicable to a wide variety of military equipment and structures, thereby eliminating volatile organic compound (VOC's) and toxic heavy metals from coatings and painting operations.

The ZDOC team of Hughes Aircraft Company, Lehigh University, University of Arizona, and the Naval Air Warfare Center Aircraft Division Warminster offers a blend of experience, expertise, and capabilities in all aspects of organic coatings technology. The ZDOC project involves research and development in several related coating development and application areas

including advanced powder, UV curable and E-coat paint development. Table 1 summarizes the major tasks and team members involved in the ZDOC project.

Table 1. Zero Discharge Organic Coatings Project Team Assignments

Task	Organizations
<b>Material Development Tasks</b>	
Corrosion Inhibitor Materials	<b>NAWCADWAR</b> , Lehigh
Powder Paint	<b>HMSC</b> , NAWCADWAR, Lehigh, HAC
UV Cure Paint	<b>HAC</b> , NAWCADWAR, Lehigh
E-Coat Paint	<b>Lehigh</b> , NAWCADWAR
<b>Applications Development Tasks</b>	
Powder Paint - IR Cure	<b>HMSC</b> , UofA
Powder Painting Non-conductive materials	<b>HMSC</b> , UofA
UV-Cure Applications Techniques	<b>HAC</b>
<b>Project Management</b>	
	<b>HMSC</b>

Note: Primary team organization for each task is listed in **BOLD**.

The task objectives for the period of January 1995 through March 1995 were as follows:

Hughes TSD: Continue testing of those photocurable resins suitable for use with non-toxic corrosion inhibiting pigments that have passed the screening goals.

Lehigh University: Continue testing to characterize the mechanisms of non-toxic corrosion inhibitors.

HMSC Tucson: Complete formulation of second iteration of inhibited powder coatings and begin testing.

NAWC: Complete formulation of second iteration of inhibited powder coatings (Same coatings as those being tested at HMSC) and begin testing. Continue evaluating methods to determine quality of conversion coated substrates.

Lehigh/NAWC: Continue development of Electrocoat process line including preparation of pigment pastes and evaluation of E-coated panels. Continue EIS testing and data analysis.

University of Arizona: Begin evaluating the effects of antistat on EMI attenuation when applied to non-conductive substrates and to determine the effect of the antistat on the barrier properties of epoxy coatings . In addition, the voids found in epoxy coatings when the antistat is used will be quantitatively evaluated based on varying initial conditions such as substrate material.

## 2.0 TECHNICAL PROBLEMS

### 2.1 Photocurable Coatings

2.1.1 Performance Property Testing. The properties most difficult to pass have been dry and wet tape adhesion to aluminum, impact flexibility, and solvent resistance. One supplier, Sokol Enterprises, supplied eight different formulations before submitting one that met all requirements, and that one met the requirements only when unpigmented. This supplier has since submitted six compositions or variations of this formulation, "Sokol 1516" with varying results when applied to six different surfaces/substrates. Based on these further results, Sokol is

reformulating again and taking the pigment more into account by pigmenting what Sokol makes with the Z-14L mix and screen testing it before submitting another formulation.

**2.1.2 Pigmentation of Clearcoats.** This contract requires the inclusion of NAWC's nonchromated corrosion inhibiting pigments and a hiding pigment. Pigmentation can interfere with the penetration of the UV energy needed for curing. Preliminary screening tests with low concentrations of each pigment separately and one mixture showed very strong absorption across the 250 to 450 nanometers range for most of the nonchromated corrosion resistant pigments.

## **2.2 Inhibitor Modeling and Characterization**

**2.2.1 Chromate Conversion Coating Evaluations.** Difficulties in obtaining good quality test coupons led to an effort to develop a method to analyze the quality of chromate conversion coating pretreatments on aluminum. Currently, the only disclosed evaluation procedure available is that specified in military specifications (mil. spec.) MIL-C-81706 and MIL-C-5541. These specifications require testing the adhesion of a standard paint over the conversion coating in question and subjecting the bare conversion coating to one or two weeks of salt fog exposure. Several deficiencies are evident in this procedure, including the duration of the tests and the inability to non-destructively test actual components.

**2.2.2 Non-Toxic Corrosion Inhibiting Pigments.** NAWC has provided non-toxic, corrosion inhibiting pigment packages for incorporation into the three coating technologies under investigation for the ZDOC program. We are currently working with the other ZDOC members and the commercial companies to determine the proper inhibitor loading levels for each resin technology. The E'coat effort is described above and the UV curable effort has not required significant feedback of information. NAWC is working closely with Hughes and the commercial companies on the powder coat materials.

The inherent hiding power of the NAWCADWAR inhibitive pigment systems is low. This characteristic allows the inhibitive pigment systems to have only a minor influence on the color of a coating. Color production thus must originate from other sources such as primary pigments. Primary pigments have high refractive indices and can impart barrier properties, however they rarely participate in active corrosion inhibition. Due to the coalescent mechanism of powder coating film formation, rheology and thus the concentration of resin and pigment solid particles influences the critical film performance of a powder coating. Solids content and rheology thus dictates the practical formulation range of powder coatings which is significantly less than the range achievable in liquid coatings.

Gloss control is an inherent problem with powder coatings. Unlike liquid coatings, the gloss of powder coatings can not be reduced by simply adding flattening agents or fillers to the formulation. Instead, micro-imperfections must be imparted to the film in order to alter the surface characteristics. This is typically accomplished by using multiple cure rate catalysts which can over cross-link the polymer matrix and produce brittleness.

Little to no work has been performed in the industry with respect to the research and development of active corrosion inhibitive powder coatings. Thus historical data is not available to aid in the present effort.

**2.2.3 Electrochemical Impedance Spectroscopy Testing.** EIS measurements were made at Lehigh University on the following systems: (1) Bisphenol A epichlorohydrin resin (Epon 828) cured with a polyamide resin (Epicure 3140) obtained from the Shell Co. at room temperature for seven days, without filters and with 0.2 micron and 5.0 micron pore diameter filters (Millipore), on chromium conversion coated (CCC) 2024 T3 aluminum panels; (2) the identical system without filters cured at 100 °C for two hours. The film thickness for both of these systems ranged between 1.8 - 2.8 mils. The electrolyte consisted of 0.01 M potassium

sulfate (decreased electrolyte concentration as reported in the previous quarterly report by a factor of ten due to concentration effects on the osmotic pressure through the film) saturated with ten selected inhibitors. These were:

- |   |   |
|---|---|
| 1. calcium strontium zinc phosphosilicate | 6. modified molybdate zinc phosphate                      |
| 2. calcium phosphosilicate                | 7. Zn salt of organic nitro compound                      |
| 3. zinc aluminum phosphosilicate          | 8. calcium modified dioxide-synthetic precipitated silica |
| 4. cinnamic acid and zinc oxide           | 9. zinc molybdate/phosphate                               |
| 5. zinc aluminum phosphate                | 10. barium metaborate                                     |

These saturated electrolyte/inhibitor solutions were prepared in distilled water and allowed to stand with occasional mixing for a period of one week at ambient temperature. The supernatant liquid was used to prepare a 0.01 M potassium sulfate. The impedance behavior of these systems has not changed in two months. There is also no discernible effect on the impedance behavior due to the filters as well as among each inhibitor system. Impedance measurements on these specimens will be continued.

Since no changes in impedance during this time period were seen, it was decided to achieve rapid failure by placing a defect in the coating. This was done by drilling an 800 micron diameter hole, with a conical point bit, through the resin film to the aluminum substrate. Using this technique, changes in low frequency impedance were seen within 48 hours among the inhibitors. For some inhibitor systems, the low frequency impedance values increased with time and with respect to the control specimen in uninhibited electrolyte. This increase in impedance is ascribed to corrosion inhibition. Other inhibitor systems exhibited no change in impedance relative to the control indicating a lack of corrosion inhibition. Additional impedance measurements on these specimens will be made.

EIS testing is continuing on both E'coat and powder coat samples at NAWC. These materials have remained in the 3.5 % NaCl for over nine months (about 7000 hours). Several of the materials have recently begun to degrade from the ideal capacitive behavior exhibited for the first 5000 to 6000 hours. The others are still exhibiting the capacitive behavior as reported for the previous quarter. Nonetheless, these tests do not provide an accelerated measure of coating degradation or substrate corrosion nor do they quickly distinguish between different coating systems. Accordingly, we are investigating additional EIS methods which can provide information more rapidly.

### *2.3 Powder Coating Non-conductive Substrates*

Much of the work to date has involved characterization of the antistats being used to render the surface of non-conductive substrates conductive. In addition, the ability to powder paint specific non-conductive substrates treated with the antistat has been investigated. During morphology and microstructure analysis of the coatings formed it was noted that voids were forming in the coating although they were not present on the surface of the coating. These voids have not effected the physical performance of the coating. Testing performed to date illustrate the voids are being caused by the polymeric substrate and the antistat, but the antistat does not cause the voids to the extent caused by the polymeric substrate. Characterization has begun to determine the effect of the substrate material on void formation and to measure the void density to insure the voids are evolving from the substrate during cure.

The effect of the antistat treatment on shielding of electromagnetic signals compared to typical conductive primers is also under investigation. This is important since many non-conductive materials are used to house electronics that require transmission of signals through the non-conductive material. The conductive primers will also be evaluated to compare the effect of void formation when a conductive primer is used versus the antistat material. In addition, the

additional weight added to the substrate when antistats are used compared to conductive primers will be documented.

The final technical issues to be evaluated are the effect of the antistat on the barrier properties of the epoxy coatings when an antistat has been used to treat a non-conductive substrate and to determine the upper limit of surface resistivity required to powder paint a non-conductive substrate.

#### *2.4 IR Curing of Powder Coatings*

The use of IR curing will be evaluated based on the physical properties of the cured powder coatings. This evaluation will insure that the properties of the paint are not effected by the IR curing when compared to the properties of the cured coatings obtained from conventional convection curing.

#### *2.5 Powder Coating Development*

The development of powder coatings has involved the development and testing of both inhibited and non-inhibited powder coatings. NAWC has performed testing on substrates coated by the supplier. HMSC has performed the powder coating of the test substrates as well as the testing on the same coating formulations being tested at NAWC. The results were compared and differences have been identified.

Based on the results from the first iteration of inhibited powder paints, a second iteration from each supplier was received. The second iteration is an attempt to improve the properties of the inhibited powder coatings.

#### *2.6 Electrocoatings Development*

**2.6.1 Establish an Electrocoat Process Line.** The development of corrosion inhibitor containing E'coat materials requires the establishment of an on site process line. Panels produced in this line must be sufficiently free of film defects to allow for proper testing. Producing panels of sufficient quality requires the development of the proper techniques in both producing the bath and operating each stage of the process. All four stages of the line, which includes metal pretreatment, electrodeposition bath, rinse and coating cure, must operate properly to produce a good quality coating.

Last quarter we reported on a problem with small defects appearing as bumps or seeds covering the panels. An improved filtration scheme removed these seeds from the bath so they would not deposit in the E'coat. However, the continuous formation of these seeds indicates that a problem exists with the E'coat bath stability.

The manufacturer suggests that less than one gram of material precipitate out of one gallon of E'coat during the pump stability test. This test continuously recycles the E'coat though the type of pump used in the full scale process line. Inside the pump is usually where the bath material is subjected to the highest shear stress and where precipitate is most likely to be formed. The magnetic stirrer we use for continuously agitating the baths subject the E'coat to much less stress than present in a pump. Despite this lower stress level our baths precipitate out more material than is acceptable for the pump stability test.

**2.6.2 Incorporate Pigments into Electrocoat Materials.** The primary task for our group is the formulation of corrosion inhibiting pigments into E'coat materials. Typically, E'coat baths are made from two separate feedstocks. Our formulation work will concentrate on introducing

different pigment dispersions into the resin paste. The resin emulsion will be used as received from the supplier.

Producing this pigment dispersion requires the development of the correct grinding technique. The equipment originally available at NAWC could not provide the required fineness of grind, which is between a 7 and a 7.5 on the Hegman gauge. We obtained several additional accessories available for our dispersion equipment. After several attempts at processing a batch of paste, these accessories worked very well at reproducing the standard BASF pigment paste formulation. Introducing corrosion inhibiting pigments into these materials is not as straight forward as processing the standard formulation. The high speed dissolver and the modified media mill require a specific range of viscosities to properly disperse the pigments into the resin. Maintaining the correct viscosity requires additions of solvent and/or water that were different for each inhibitor.

Once the inhibitor containing pastes are produced E'coat baths are assembled in the usual manner. Besides evaluating the electrocoated panels for the usual performance properties, we must consider the compatibility of the inhibitors with the E'coat materials. Any incompatibilities will cause the bath to precipitate material at an accelerated rate. Finally, the yield from each batch of paste was very poor usually less than 50%. Although some loss is unavoidable due to entrapment on the grinding media and testing samples during processing, this amount of loss was excessive.

**2.6.3 Obtain Access to Anodic Electrocoat Materials.** All formulation work performed to date used a single cathodic E'coat material as the basis for the formulation work. The automotive industry prefers cathodic materials because they form a better barrier and so provide improved corrosion protection. However, the ZDOC program may benefit from the slightly reduced barrier properties provided by anodic E'coat materials. The reduced barrier properties may allow the corrosion inhibiting pigments to leach out into any defect areas and so provide protection. Cathodic materials may be such good barriers that the inhibitors are encapsulated in the coating so tightly that they are unable to leach out to provide protection.

A non-disclosure agreement is in place between NAWC and PPG. NAWC provided PPG with their proprietary, corrosion inhibiting, pigment formulations. PPG will determine which packages appear to offer the best chance of compatibility with their E'coat materials, primarily their anodic products. NAWC also requested that PPG provide samples of their E'coat for our in-house process line. However, PPG appears unwilling to provide this level of support so, the NAWC E'coat process line may be limited to formulating with cathodic materials for the duration of the ZDOC program.

## 3.0 GENERAL METHODOLOGY

### 3.1 Photocurable Coatings

**3.1.1 Screening Testing on Photocurable Coatings.** During this quarter, testing continued on the most promising materials evaluated to date. In addition another contract was issued to the Naval Surface Warfare Center (NSWC) to continue promising work being performed by Barbara Howell on low toxicity, UV curable paints for use on submarines. The suppliers and the candidate materials on which testing is continuing are listed in Table I in Appendix A.

**3.1.2 Incorporation of Non-toxic Corrosion Inhibiting Pigments.** Although all ingredients for each of NAWC's corrosion inhibiting pigment formulations have been obtained from the manufacturers, only the Z-14L mix has been used this quarter. The UV transmission of

each of these NAWC pigments have been obtained when mixed in a glycerol base. Incorporation of the nonchromated corrosion inhibiting pigments was begun last year. During the screening phase, most of the candidate materials were applied to the Mil-C-5541 conversion coated 2024 T3 and TO aluminum baseline substrate and also, this quarter, to five other substrates (zinc phosphated 1010 steel, zinc phosphated plus MIL-C-8841 wash primed 1010 steel, zinc phosphated, MIL-C-8841 wash primed and MIL-P-23377 primed 1010 steel, bare TO and T3 aluminum, and chromate conversion coated plus MIL-P-23377 primed TO and T3 aluminum). These materials will be tested for coating properties. Formulations will be optimized and put into final testing based on the results obtained during this phase.

### *3.2 Inhibitor Modeling and Characterization*

**3.2.1 Modeling.** In an effort to understand the mechanisms of corrosion inhibition in an intact coating in a short period of time, relative to the intact 828/3140 system, it was decided to select a resin system that would be more permeable to water than the 828/3140 system. This system consisted of a 1:1 weight ratio of a bisphenol A epoxide resin (DOW DER 332) and a polyglycol diepoxyde resin (DOW DER 736) cured with a polyamide resin (UNI-REZ 2140 obtained from Union Camp Co.) at 100°C for 2 hours. These systems were made with selected inhibitors: (1) incorporated in the 736 resin (0.3% by weight), (2) saturated in the electrolyte, and (3) in both the 736 resin and the electrolyte. The film thicknesses of these coatings ranged from 1.0 - 1.5 mils. Impedance measurements on these systems showed that the impedance of these coatings was lower than the 828/3140 coatings, but insufficiently low enough to see any changes in the low frequency impedance due to inhibitor effects. In order to expedite any impedance changes, it was decided to create a defect in the coating in the same manner as described previously. These tests are currently in progress; however, conclusive results have yet to be obtained.

Since the 736/332/2140 systems did not show any low frequency impedance changes as intact films, it was decided to select an even more permeable coating to water. The system selected was the 736 resin cured with 2140 for 2 hours at 100°C (film thickness was between 0.5 - 1.0 mils). The impedance of this coating system was found to be lower than the 736/332/2140 system, but major changes in the low frequency impedance have not been observed up to the present time (4/7/95). It was concluded that the impedance results on the intact coating systems on CCC aluminum substrates may have been affected by the presence of the conversion coating. Therefore, two approaches were implemented to test this hypothesis: (1) the use of bare aluminum panels as substrates, and (2) the use of a more aggressive electrolyte with the CCC substrates.

Two surface treatments were employed on the bare aluminum panels: (1) as -received panels immersed for 30-60 seconds in hot (120-140°F) 5% NaOH, etched 1-2 minutes in 180°F 25% sulfuric acid, rinsed in distilled water for 30-60 seconds, immersed in room temperature 50% nitric acid for 60-90 seconds, rinsed with distilled water, washed with ethanol, allowed to air dry at room temperature, and then placed in a silica gel dessicator; and (2) as-received panels rinsed with acetone and wiped with lintless laboratory tissue. These panels were then coated with the 828/2140 formulation cured at room temperature for 7 days and with the 736/2140 formulation cured for 2 hours at 100°C.

The more aggressive electrolyte chosen was 0.01 M potassium sulfate plus 0.003 M potassium chloride. This electrolyte was saturated with various inhibitors by adding an excess of inhibitor to the electrolyte solution and supernatant liquid was used as the electrolyte/inhibitor solution.

Impedance measurements on the 736/2140 system on bare aluminum were made with the inhibitor: (1) incorporated in the 736 resin (17% by weight), (2) saturated in the electrolyte (both 0.01 M potassium sulfate and 0.01 M potassium sulfate plus 0.003 M potassium chloride

solutions), and (3) in both the coating and the electrolyte. Initial results showed that: (1) the low frequency impedance of the 736/2140 coated CCC panels was 1 to 2 orders of magnitude higher than the 736/2140 coating on bare aluminum, and (2) the low frequency impedance changed with time (24 hours) and with respect to the control. These results demonstrate that by using this method (a simple impedance cell and a porous coating on the CCC panels), the impedance of CCC panels can be determined; and therefore, can be evaluated as a corrosion preventative. Impedance tests are also in progress using the 828/3140 system on CCC, chemically cleaned, and solvent washed aluminum panels with an 800 micron diameter defect.

**3.2.2 Chromate Conversion Coating Evaluation.** As a first step several candidate testing procedures were selected by searching the literature and on the advise of in-house NAWC workers. Each method selected was evaluated with respect to its ability to distinguish between conversion coated aluminum samples of low (below mil. spec.), middle (within mil. spec.) and high (above mil. spec.) coating weights. Those methods surviving the first round of the study are to be further investigated. The samples for this study are produced at NAWC specifically for each round of the investigation.

**3.2.3 Electrochemical Impedance Spectroscopy.** Commercially available E'coat and powder coat materials are investigated and characterized to provide background information. The work is extended to developmental coatings and/or model systems where appropriate. The characterization of a coating is approached by first mathematically modeling the impedance spectra using equivalent circuits. Then, plausible physical models are tested by measuring the effect on the impedance spectra of varying testing parameters.

### *3.3 Powder Coating Non-conductive Substrates*

In order to determine the effects of both the antistat materials and typical conductive primer on EMI signals, an RF generator and detection equipment will be used to measure the attenuation of signals through a 2 in. X 2 in. substrate. The epoxy substrates will be treated with antistat and the attenuation will be measured and compared to substrates treated with conductive primer and to a clean untreated epoxy substrate.

Electrochemical Impedance Spectroscopy will be used to analyze the effects of antistat on the barrier properties of epoxy coatings when antistat has been used to pretreat the substrate surface. This work will insure that the antistat does not have any effect on the barrier properties of the epoxy coating.

An image analyzer will be used to measure the percentage of area (cross section) of the epoxy coating covered with voids, the average size of the void, and the average thickness of the epoxy coating. This will allow for determination of the major cause of void formation within the epoxy coating and if a conductive primer can act as a barrier to prevent volatiles from escaping from the substrate and into the epoxy coating.

### *3.4 IR Curing of Powder Coatings*

The physical properties of powder coatings cured by IR will be evaluated using the test plan for the inhibited powder coatings. This testing will be performed during the last quarter of the program.

### *3.5 Powder Coating Development*

The test plan for development of inhibited powder coatings development has been documented and reported in earlier reports. This test plan was used by both NAWC and HMSC to test the powder coatings formulations under evaluation. The first iteration of powder coatings

testing has been completed. The test results have been compared and documented. The second iteration of inhibited powder coatings have been formulated and testing has begun. The second iteration is intended to improve the properties of the powder coatings obtained in the first iteration. The same test plan used for the first iteration will be employed to test the second iteration.

### *3.6 Electrocoat development*

Before any formulation work can begin we must develop the techniques and the industrial art for operating an E'coat process line. An in house effort formulating electrocoat materials containing non-toxic, corrosion inhibiting pigments is a multistep process.

First, each stage of the process line must operate properly to produce a good quality coating. Once the process line is operating properly with commercially available materials we may then begin to produce our own feed materials. Our formulation work will concentrate on introducing different pigment dispersions into the resin paste.

A typical E'coat bath is made from two separate feedstocks. The bulk of the resin is supplied to the bath as an emulsion that does not contain any pigments. The pigments, whether for color or corrosion protection, are introduced to the bath as a separate feed stream. This stream consists of the pigments and other powder additives dispersed throughout additional resin.

Producing this pigment dispersion requires the development of the correct grinding technique. This technique consists of more than assembling the correct equipment and turning it on to mix. These dispersions require the correct viscosity and flow pattern during the dispersion process. Additions of the pigment and later of water or other solvent to aid the process are best determined by previous experience. Once the techniques for producing the standard pigments paste are developed other corrosion inhibiting pigments may be attempted.

## **4.0 TECHNICAL RESULTS**

### *4.1 Photocurable Coatings*

**4.1.1 Supplier Summary.** Table I, shown in Appendix A, shows the candidates still in consideration. Seven other tables, shown in Appendix A, show the latest screening test results of the candidate materials submitted for consideration.

**4.1.1.1 Sokol Enterprises Photocurable Coating.** Sokol Enterprises has been developing a line of coatings which are 100% solids and are being targeted for the automotive industry. The very low (sprayable) viscosity of this material makes them particularly unique among candidates evaluated to date. Of the nine different formulations submitted by Sokol Enterprises, the best candidate, Sokol 1516, was made into six different composition variations and each pigmented 10% with pigment formulation Z14L. Then each of the six composition variations was applied to each of the six types of substrates being studied. A full set of the screening tests was run on all these combinations and the results shared with Sokol Enterprises. Sokol learned much from how each composition variation affected the screening test results and now knows to add some soft urethane acrylate and 10% Halox additive to arrive at the final formulation. Sokol will try out with the Z14L pigment mix on aluminum and steel substrates and, if it satisfactorily at that point, will provide this final formulation to HAC-TSD for final testing. Hughes has been working closely with Sokol to provide some formulation guidance toward improving the performance properties of the 1516 product.

**4.1.1.2 3M Dual Cure Coating.** The 3M Corporate Research Lab has been working on a dual cure, photocurable, coating for several years under a DOE project (Contract No. AC04-

88ID12692). The goals of this program were similar in many ways to the ZDOC program with several exceptions. 3M was evaluating a primer/topcoat system and was not evaluating a nonchromated corrosion inhibiting primer. The 3M submittal is a urethane/acrylate formulation with titanium dioxide pigment (designated "3M Dual Cure Polyurethane Coating") and contains 340 g/l VOC. A clearcoat was also submitted to enable incorporation of the pigments used in this program. One of the ingredients of the formulation is sensitive to shear stresses and was not able to survive ball milling. Therefore, 3M submitted a three part kit to avoid this problem. Ball milling was unsuccessful due to the high viscosity of the grind resin. This problem was overcome by adding a highly evaporative solvent to adjust the viscosity of the grind resin, using the ball mill to pigment it and evaporating off the solvent afterwards. A Hegman grind of 6 1/2 was obtained at 25% pigment loading. However, due to time and manpower constraints, this coating has not at this date been applied to substrates for screen testing; that is expected to occur the first week of April.

3M representatives had previously agreed to continue work on their formulation to further reduce the VOC content. The formulation includes VOC's in a letdown solvent (which was not included in the unpigmented formulation submitted) and also in a resin component. Since the DOE contract was completed, 3M has decided not to continue the search for a solvent-free resin. 3M also has elected not to release the formulation preventing Hughes from continuing the effort. The VOC of the existing formulation is slightly over 120 g/l which is a significant improvement over the current 340 and 420 g/l paints in industry.

**4.1.1.3 Dymax Corporation.** The Dymax formulation with the most promising properties is their X256-16-1 as stated in the previous quarterly report. It is now known by their commercial designation "Multicure 984-TC" trigger cure conformal coating. It is based on UV curable conformal coating technology for electronic circuit boards. This urethane/acrylic copolymer cures in the UV light and in shadows with a secondary cure mechanism using atmospheric moisture. This formulation was pigmented to 25% with the Z14L pigment mix and screen tested with generally good results.

**4.1.1.4 Herberts.** Herberts, Inc. is a producer of powder coatings and is working with HAC-MSG in the powder phase of this program. Herberts also has produced a unique formulation that fuses at an ultra low 160 °F and is UV curable. This combination of properties could be extremely useful for temperature sensitive substrates that cannot withstand the 250 to 350 °F fusion temperatures of most commonly seen low temperature powders. Initial submittals did not meet impact flexibility requirements. In this past quarter several other submittals again were deficient in flexibility. Kevin Biller of Herberts had been attempting to improve flexibility by adding plasticizers and flexibilizers to the Ciba-Geigy resin system he was using. During this quarter he worked with European companies associated with Herberts and settled on a different base resin system. He was somewhat successful with one submitted by Herberts' Switzerland headquarters but is still fighting solvent resistance when he makes it flexible enough to have good impact and 1/4 inch mandrel flexibility. He completed a round robin of testing this formulation (UV Cure #30) at different labs including one in Sweden. He will be supplying cured panels with this formulation on three different kinds of substrates furnished by TSD: bare aluminum, chem filmed aluminum and zinc phosphated steel, by the first of April for our final testing.

**4.1.1.5 NSWC (Naval Surface Warfare Center).** Barbara Howell of NSWC continued her work on UV curable paints under TSD direction. Because flexibility had been a problem, we had encouraged her use of aliphatic urethane acrylate formulation technology but kept having problems with ingredient incompatibility which caused lumps and incomplete cure in the coating. It was decided instead to focus on her previous epoxy acrylate formulation which had previously worked fairly well on steel substrates. Also, she is especially interested in developing a paint for maintenance of shipboard equipment which is mainly steel. Therefore, in a

procurement contract let in early March, it was agreed that she would furnish the epoxy acrylate formulation, improving it as necessary, sending 200 ml of it to us to pigment and apply, and applying it herself to 12 grit blasted steel panels and 12 "tight rusted" steel panels. These two kinds of steel surface preparation are what the Navy does aboard ship before painting. "Tight rusted" steel is rusted steel that has been wire brushed to remove all loosely adhering matter.

In correspondence dated March 31, 1995, Barbara Howell said she is sending the panels cured with a V-bulb and belt speed of 152 ft/min as follows:

Package A: 12 grit blasted steel panels with three coats of epoxy acrylate containing about 20% pigment. Warpage from the grit blasting, however, made it difficult to get a smooth coating with the #20 rod that was used.

Package B: 12 tight rusted steel panels with the same coating as above except containing about 30% pigment. Bumpiness of the rust, however, made it difficult to get a smooth coating.

Package C: 12 zinc phosphated steel panels with the same coating as for Package B and smoothed with the #20 rod.

**4.1.2 Screening Test Results.** The screening tests selected and test results are included in the tables attached in Appendix A. These tests were performed in-house on specimens coated 2-3.5 mils thick.

## *4.2 Inhibitor Modeling and Characterization*

**4.2.1 Modeling.** Initial results have confirmed the viability of the experimental design and work is underway in evaluating selected inhibitors. To date it has been shown that (1) 828/3140 resin systems cured at room temperature and 100°C without defects showed no change in low frequency impedance up to 2 months, (2) the same system with an 800 micron diameter defect showed changes in low frequency impedance within 48 hours and changes among individual inhibitors with respect to the control, (3) more water permeable coating systems (736/332/2140 and 736/2140) on CCC panels showed little change in low frequency impedance, and (4) 736/2140 coatings on bare aluminum substrates showed changes in low frequency impedance within 24 hours and changes with respect to the control.

The PPG/Lehigh non-disclosure agreement has been signed by both parties.

**4.2.2 Chromate Conversion Coating Evaluation.** This quarter we completed the test plan for the second round of the project investigating alternate methods of analyzing the quality of chromate conversion coating pretreatments on aluminum. This second round will analyze several different factors. A total of five chromate conversion coating weights will be produced and two different conversion coating materials will be available. Also for those methods which can provide justification, two types of deoxidization, one chemical and one mechanical, are available. We are awaiting a new batch of bare panels to arrive before continuing with the project.

**4.2.3 Electrochemical Impedance Spectroscopy Testing.** During the past quarter we continued to characterize electrodeposited coatings using impedance spectroscopy. A physical model for E'coatings exposed to SO<sub>2</sub> salt spray was sought using the approach outlined at the January 1995 contractors' meeting.

The number of apparent time constants observed is a function of bias potential. This quarter it was discovered that the true number of time constants remains constant while the values of some of the circuit elements change.

Progress was made in relating the impedance spectra of the uncoated pre-treated aluminum to the E'coated pre-treated aluminum. In particular, E'coats on sulfuric acid anodized aluminum have

been studied. It may be difficult to extend this approach to chromated surfaces, but an attempt will be made. This work will help in understanding the observation of better SO<sub>2</sub> salt spray performance for sulfuric acid anodized versus chromate conversion coated E'coats.

#### *4.3 Powder Coating Non-conductive Substrates*

The results of the work performed during the first quarter 1995 will not be completed until the second quarter 1995.

#### *4.4 IR Curing of Powder Coatings*

The testing of inhibited powder coatings will be completed the second quarter 1995.

#### *4.5 Powder Coating Development*

New NDA's were prepared and executed in order to continue the working relationship with Herberts on powder coating technology and with BASF on E-coat technology. Long term (2000 hr) salt fog corrosion resistance testing of the first set of inhibited powder coatings from Herberts and Morton on aluminum was completed. The corrosion inhibitive pigments appear to have a positive influence on the salt fog corrosion resistance performance on aluminum. Accelerated weathering testing of the first set of inhibited powder coatings is near completion with an expected termination date of 12 April 95. Met with Hughes (Tucson) to plan the powder coating development activities for the final phase of the ARPA effort. The Herberts epoxy and the Morton epoxy-phenolic with the uninhibited, inhibited, and mixture (2:1) pigment systems will be formulated at a PVC of 20% and 15% respectively. Testing is underway on these formulations.

NAWCADWAR and Hughes will stop powder coating laboratory testing on 1 May 95. By 2 June 95, NAWCADWAR and Hughes will mutually consolidate and integrate the powder coating data and discussion sections into a version appropriate for the final report coordination by Jim Leal.

#### *4.6 Electrocoat development*

**4.6.1 Electrocoat Process Line.** Last quarter we reported on a problem with small defects or seeds appearing in the E-coat film. A better filtration scheme prior to each panel processing session vastly improved the coating quality almost to the level of the Mac-Air panels. Some slight difference between our coatings and theirs is expected since they use continuous filtration for their 100 gallon tank. However, the continuous formation of these seeds or of other precipitated material indicates that a problem exists with the long term stability of the E-coat bath.

The stability of the bath containing the standard formulation pigment paste does not meet the manufacturer's requirements. The manufacturer suggests that less than one gram of material precipitate out of one gallon of E-coat during a pump stability test. Baths made from pigment paste produced at NAWC and produced by BASF could not meet this requirement during agitation by a magnetic stirrer.

The long term stability problem may be caused by an insufficient amount of acid available for the E-coat resin. The amount of available acid controls the degree of resin solubilization, called total neutralization (TN%) by the E-coat industry. This factor must be determined for individual baths. Measuring the pH does not provide an accurate representation of the TN% character of the bath material. To properly determine the TN% requires a potentiometric titration for both the acidic and basic milliequivalents, MEQa and MEQb respectively. We have established the

procedures necessary to perform the non-aqueous, potentiometric titrations to determine the total neutralization of the resin in E'coat materials.

Our evaluations of the baths for total neutralization found that the MEQa was slightly below the optimum value but not enough to account for the stability problem. After consolations with BASF it was decided that the resin emulsion might be out of specifications. While we were waiting for new materials to arrive, they were delayed due to a reformulation of the grinding resin, we noticed that the old emulsion had taken on a slight pink cast. This usually indicates contamination by bacteria which would cause a stability problem similar to the one we observed.

**4.6.2 Electrocoat Pigment Dispersions.** Additional accessories are available for the Dispermat high speed dissolver that allows us to grind the pigment paste in the same manner as BASF. After several attempts at processing a batch of paste, these accessories worked very well at reproducing the standard BASF pigment paste formulation. However, introducing the corrosion inhibiting pigments into these materials is not as straightforward as processing the standard formulation.

The high speed dissolver and the modified media mill used to obtain the desired fineness of grind require a specific range of viscosities to operate. If the mill base viscosity falls outside the proper range the dispersion process will not proceed and the resin may even undergo degradation. Maintaining the correct viscosity while dispersing the inhibitive pigments requires additions of butylcellosolve solvent and/or water. These additions differ in quantity and order for each of the different corrosion inhibitors.

Six corrosion inhibiting pigments are used in assorted combinations to form the five NAWC proprietary pigment packages. Five of these inhibitors, SZP-391, MW-101, Phos Plus, KW-84, and Sicron-RZ have been incorporated into pigment pastes. The ZMP inhibitor supply at NAWC was inadvertently discarded so the sixth pigment used in the NAWC proprietary inhibitor packages could not be produced in a timely fashion. Four of the five pastes were based on the standard BASF formulation with 75% by weight of the TiO<sub>2</sub> replaced with one of the inhibitors.

The SZP-391 pigment appeared to grind in the normal manner, however, after two days the paste gelled into a solid mass. The MW-101 proved difficult to grind as an addition of 10g of solvent into the 400g batch of paste was required to complete the process. Surprisingly, this paste still remains a liquid with approximately the correct viscosity. The Phos Plus also proved difficult to properly grind. Although only a small amount of additional solvent was required, the rheological behavior of the paste during the grinding was wrong. This paste exhibited pseudoplastic behavior with a very high yield point during the grinding and when checked after one week this paste also had gelled. Processing the KW-84 pigment went smoothly although maintaining the correct viscosity required several solvent additions. This paste also remains a liquid with approximately the correct viscosity.

The Sicron-RZ was the pigment which proved to be the most trouble to properly grind. This pigment has a low specific gravity and a high oil absorption which may make higher loading levels difficult to disperse. Our first attempt replaced only 50% by weight of the TiO<sub>2</sub> with this inhibitor into the standard formulation. The pigment was hard to cut in and eventually the paste completely gelled. Additions of water and solvent could not recover this batch. To salvage the ZrO<sub>2</sub> beads the entire batch was discarded.

This first attempt possibly failed because the volume of dry components exceeded the wetting capacity of the grind resin. The second attempt at producing this paste incorporated 25 % by weight of the usual amount of TiO<sub>2</sub> as Sicron-RZ and 25 % of the usual amount of curing catalyst. At this significantly reduced loading level the processing went smoothly although maintaining the correct viscosity required several solvent additions. This paste also remains a liquid with approximately the correct viscosity.

Finally, we were having a problem with the excessive loss of material as the average yield was less than 50% even with the standard formulation. Although some loss is expected due to sample testing and separating the beads, this amount of loss was excessive. When we measured the viscosity of the standard paste it was twice the suggested value. This indicates that the pastes were not being letdown sufficiently. Most of the pastes made prior to this analysis, including the first three inhibited pastes, were probably at too high a viscosity. The newest batch of standard paste was subsequently letdown to the correct viscosity and the KW-84 and Sicron-RZ pastes were letdown properly the day they were produced.

**4.6.3 Electrocoat Baths Containing Corrosion Inhibitors.** All five of the corrosion inhibitor containing pigment pastes were used to make working E'coat baths. Each inhibitor was added to a separate bath so that no combinations were evaluated. Four of the inhibitor pastes contain the full package of inhibitor and other pigments and additives. These single inhibitor containing baths were made from a single pigment paste. However, due to the difficulties of grinding the Sicron-RZ this bath had to be made from two pastes. The total weight of paste added to the Sicron-RZ bath was the same as for all other inhibitor containing baths. We combined 75% of the standard formulation paste produced at NAWC with 25% of the Sicron-RZ paste.

Each of these inhibitor containing baths were prepared on a separate day. After 24 hours of stirring, the bath is filtered into a clean vessel and then brought to the desired operating temperature. A voltage ladder determines the correct voltage for the desired film thickness for each inhibited bath. A total of 20 3x4 inch panels were coated from each bath.

The bath non-volatile content and the pigment to binder ratio are monitored to check the bath stability. Bath stability does not meet the manufacturer's requirements for any of the inhibitor containing baths. All five inhibitor containing baths lose approximately a gram of material from a one liter bath during overnight stirring. This amount is much more than is lost from the standard formulation bath made from the contaminated batch of resin emulsion.

Performance evaluations are underway on the E'coated panels which contain inhibitors. These panels and panels coated by Mac-Air, for use as controls, are now in neutral and SO<sub>2</sub> salt fog exposure cabinets. They all pass water resistance and tape adhesion tests. The other evaluation procedures, most notably chemical resistance, will soon be underway. Also, we submitted a sample panel from each inhibitor containing bath to the analytical lab for atomic absorption analysis of the film.

## 5.0 IMPORTANT FINDINGS AND CONCLUSIONS

### 5.1 Photocurable Coatings

5.1.1. A follow-on contract has been granted to Barbara Howell of NSWC to continue her work on UV curable paints based on her previous work which had resulted in a Navy gray, epoxy based paint cured with a xenon lamp for touch-up painting on a submarine. She has submitted 200 ml of an improved version of the epoxy formulation to apply pigment and apply to selected substrates for final testing. She will pigment the same formulation to between 20 and 30% of Z14L and apply and cure it on various steel substrates. When these painted panels are received they will be included in the final testing.

5.1.2. The Dymax 984-TC, pigmented to 25% Z14L, provided somewhat satisfactory screening test results and will be included in the final testing pending some improvement that needs to be made to its flexibility.

5.1.3. Herberts is continuing to submit UV cured powder paints for testing. A new resin system will be used to improve impact resistance. Kevin Biller at Herberts has steel and aluminum panels which he will coat with the final formulation and send them to TSD completely cured for inclusion in the final testing.

## *5.2 Inhibitor Modeling and Characterization*

5.2.1. Variation of EIS parameters such as electrolyte and electrode bias potential shows promise for determining physical models from the resulting effect on the impedance spectra.

5.2.2. The difference between the number of apparent and actual time constants observed with EIS has been clarified for a number of E'coated systems.

5.2.3 Work is continuing on relating the impedance spectra of the uncoated pretreated aluminum to the E'coated pretreated aluminum. This will help our understanding of the different corrosion performance in SO<sub>2</sub> salt spray observed between E'coated panels with different pretreatments.

5.2.4 NAWC and Lehigh University prepared a test plan for the second round of the project analyzing the quality of chromate conversion coating pretreatments on aluminum. Additional bare aluminum panels for use in this effort were recently ordered.

## *5.3 Powder Coating Development*

5.3.1 Long term (2000 hr) salt fog corrosion resistance testing of the first set of inhibited powder coatings from Herberts and Morton on aluminum was completed. The corrosion inhibitive pigments appear to have a positive influence on the salt fog corrosion resistance performance on aluminum.

5.3.2 Met with Hughes (Tucson) to plan the powder coating development activities for the final phase of the ARPA effort. NAWCADWAR and Hughes will stop powder coating laboratory testing on 1 May 95. By 2 June 95, NAWCADWAR and Hughes will mutually consolidate and integrate the powder coating data and discussion sections into a version appropriate for the final report coordination by Jim Leal.

5.3.3 New NDA's were prepared and executed in order to continue the working relationship with Herberts on powder coating technology and with BASF on E-coat technology.

## *5.4 Electrocoat Development*

5.4.1 Work is continuing on the establishment of an in-house E'coat process line. The E'coats produced in this line are nearly as free of imperfections as those produced by Mac-Air in their 100 gallon tank. However, the bath containing the standard BASF formulation does not meet the specified stability requirements. We believe that this problem may be traced to a contaminated batch of the resin emulsion.

5.4.2 Produced five pigment pastes containing individual corrosion inhibiting pigments. The five inhibitors selected were those used to make the NAWC proprietary pigment packages including MW-101, SZP-391, Phos Plus, KW-84, and Sicron-RZ. The ZMP inhibitor supply at NAWC

was discarded so the sixth pigment used in the NAWC proprietary inhibitor packages could not be produced in a timely fashion.

5.4.3 Mixed working E'coat baths using the above five inhibitor containing pigment pastes. A voltage ladder determined the correct voltage for the desired film thickness for each inhibited bath. Also, the bath non-volatile content and the pigment to binder ratio are monitored to check the bath stability. A total of 20 panels were coated from each bath.

5.4.4 Began performance evaluations on the E'coated panels which contain inhibitors. These panels and panels coated by Mac-Air, for use as controls, are now in neutral and SO<sub>2</sub> salt fog exposure cabinets. They all pass water resistance and tape adhesion tests. The other evaluation procedures, most notably chemical resistance, will soon be underway.

5.4.5 Began investigating other possible corrosion inhibitors for use in the E'coat materials. These additional inhibitors will constitute the final iteration for the ZDOC E'coat formulation work. I am specifically interested in inhibitors designed for systems whose stability is sensitive to bivalent ions. Several orders have been placed and I expect to order some additional samples after consulting with NAWC and Lehigh.

5.4.6 Under a non-disclosure agreement PPG attempted to incorporate the NAWC non-toxic, corrosion inhibiting, pigment packages into their anodic E'coat. PPG was unable to produce a stable E'coat bath with any of the inhibitor packages. They did not submit any coated panels for performance testing and may not have successfully coated any panels.

## **6.0 SIGNIFICANT HARDWARE DEVELOPMENTS**

N/A

## **7.0 SPECIAL COMMENTS**

N/A

## **8.0 IMPLICATIONS FOR FURTHER RESEARCH**

N/A

## **9.0 REFERENCES**

N/A

# **ZERO DISCHARGE ORGANIC COATINGS**

## **Powder Paint - UV Curable Paint - E-Coat**

### **Appendices**

**Appendix A**

**UV Curable Coatings -Test Results**

## **APPENDIX A**

### **UV Curable Coatings**

#### **Test Results**

## UV CURABLE COATING CANDIDATES EXPOSED TO SCREENING TESTS (ZDOC PROGRAM)

Material & manufacturer	Material Type	Appx. viscosity (cps)	Specific gravity	VOC content (g/l)	Pigmented?	Comments
Sokol 15XX series	urethane/acrylate	300	TBD	0	no	specialty formulations. 1516 is leading Sokol candidate. It is being modified for final testing
3M 631 clear	urethane and acrylic	1000	1.2	120	no	Three component. From DOE contract. Ball mill pigmented with solvent thinner while ballmilling
NSWC epoxy	epoxy	TBD	TBD	0	gray	Supplied pigmented with Z-14L
Dymax 984 TC	urethane/acrylate	150	1.05	0	no	modified conformal coating
Herberts UV powder	# 30 epoxy	N/A	1.96	0	white	Unique UV curable, powder coating with a very low, 200 °F fusion temperature. A new formulation based on more flexible resin chemistry.

Table I. Suppliers and Candidate Materials on Which Testing is Continuing

# Zn-Ph 1010 Steel

<u>UV COATING</u>	<u>MEK RESISTANCE</u>	<u>PENCIL HARDNESS</u>	<u>DRY ADHESION</u>	<u>WET ADHESION</u>	<u>IMPACT, FORWARD, FT-LBS</u>	<u>IMPACT, REVERSE, FT- LBS</u>	<u>CONICAL MANDREL</u>	<u>GLOSS</u>
SOKOL 1510, Z14L:10%pvc	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good	Good	10	<2	Fail	67.3
SOKOL 1511, Z14L:10%pvc	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good	Good	14-18	<2	Fail	75.9
SOKOL 1513, Z14L:10%pvc	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good	Good	10	<2	Fail	73.3
SOKOL 1514, Z14L:10%pvc	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good	Good	8	<2	Fail	33.5
SOKOL 1515, Z14L:10%pvc	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good	Good	10	<2	Fail	74.6
SOKOL 1516, Z14L:10%pvc	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good	Good	12	<2	Fail	63
3M 631								
DYMAX 984 TC, Z14L:25%pvc	Good	9H	Good	Good	0	0	Fail	46.6

# Zn-Ph 1010 Steel + Wash Primer

<u>UV COATING</u>	<u>MEK RESISTANCE</u>	<u>PENCIL HARDNESS</u>	<u>DRY ADHESION</u>	<u>WET ADHESION</u>	<u>IMPACT FORWARD, FT-LBS</u>	<u>IMPACT REVERSE, FT- LBS</u>	<u>CONICAL MANDREL</u>	<u>GLOSS</u>
SOKOL 1510	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good@UV/primer Fail@ primer/ steel	Good	14	<2	Fail@Primer/ steel interface	5.6
SOKOL 1511	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good@UV/primer Fail@ primer/ steel	Good	8	<2	Fail@Primer/ steel interface	64.5
SOKOL 1513	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good@UV/primer Fail@ primer/ steel	Good	18-22	2	Fail@Primer/ steel interface	74.8
SOKOL 1514	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good@UV/primer Fail@ primer/ steel	Good	8-10	<2	Fail@Primer/ steel interface	30
SOKOL 1515	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good@UV/primer Fail@ primer/ steel	Good	14	0	Fail@Primer/ steel interface	79.4
SOKOL 1516	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good@UV/primer Fail@ primer/ steel	Good	8	<2	Fail@Primer/ steel interface	58.3
3M 631								
DYMAX 984 TC	Good	9H	Good	Good	0	0	Fail	52.6

# Zn-Ph 1010 Steel + Wash Primer + Mil-P-23377

<u>UV COATING</u>	<u>MEK RESISTANCE</u>	<u>PENCIL HARDNESS</u>	<u>DRY ADHESION</u>	<u>WET ADHESION</u>	<u>IMPACT FORWARD, FT-LBS</u>	<u>IMPACT REVERSE, FT-LBS</u>	<u>CONICAL MANDREL</u>	<u>GLOSS</u>
SOKOL 1510	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good @ UV/primer Fail @ primer/ steel	Fail	20	<2	Good	57.8
SOKOL 1511	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good @ UV/primer Fail @ primer/ steel	Fail	34	4	Fail @ Primer/ steel interface	71.4
SOKOL 1513	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good @ UV/primer Fail @ primer/ steel	Fail	34	2	Good	75.8
SOKOL 1514	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good @ UV/primer Fail @ primer/ steel	Fail	30	2	Good	60.8
SOKOL 1515	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good @ UV/primer Fail @ primer/ steel	Fail	38	6	Good	72.8
SOKOL 1516	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good @ UV/primer Fail @ primer/ steel	Fail	16-18	<2	Good	64.3
3M 631								
DYMAX 984 TC		9H	Good	Good	12	2		

# Aluminum, Bare, 2024, T0

<u>UV COATING</u>	<u>MEK RESISTANCE</u>	<u>PENCIL HARDNESS</u>	<u>DRY ADHESION</u>	<u>WET ADHESION</u>	<u>IMPACT FORWARD, FT-LBS</u>	<u>IMPACT REVERSE, FT-LBS</u>	<u>CONICAL MANDREL</u>	<u>GLOSS</u>
SOKOL 1510	Gloss Loss-5 rubs: Thk Loss-40 rubs	9H	Good	Fail	32	2	Pass	98.7
SOKOL 1511	Gloss Loss-5 rubs: Thk Loss-40 rubs	9H	Good	Fail	8	<2	Pass	98.1
SOKOL 1513	Gloss Loss-5 rubs: Thk Loss-40 rubs	9H	Good	Fail	6	<2	Pass	113.9
SOKOL 1514	Gloss Loss-5 rubs: Thk Loss-40 rubs	9H	Good	Fail	4	<2	Fail	79
SOKOL 1515	Gloss Loss-5 rubs: Thk Loss-40 rubs	9H	Good	Fail	6	<2	Pass	113.5
SOKOL 1516	Gloss Loss-5 rubs: Thk Loss-40 rubs	9H	Good	Fail	14	2	Pass	84.6
3M 631								
DYMAX 984 TC	Good	9H	Good	Fail	4	0	Fail	49.3
HERBERTS ULTRAFAST WHITE 200F+UV	Gloss Loss-1 rub			Good	32	2		

## UV RESULTS, BARE ALUMINUM

HERBERTS ULTRAFAST WHITE 260F+UV	Gloss Loss-1 rub		Good	26	2	
HERBERTS UV CURE#30, 1.1-1.4MILS	Gloss Loss-1 rub		Good	36	26	
HERBERTS UV CURE#30 1.2-1.5MILS	Gloss Loss-1 rub		Good	34	18	
HERBERTS UV CURE#30 1.6-1.9MILS	Gloss Loss-1 rub		Fail	16	6	

# Aluminum, Conversion Coated

<u>UV COATING</u>	<u>MEK RESISTANCE</u>	<u>PENCIL HARDNESS</u>	<u>DRY ADHESION</u>	<u>WET ADHESION</u>	<u>IMPACT FORWARD, FT-LBS</u>	<u>IMPACT REVERSE, FT-LBS</u>	<u>CONICAL MANDREL</u>	<u>GLOSS</u>
SOKOL 1510	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good	Good	6	<2	Pass	65.8
SOKOL 1511	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good	Good	6	2	Pass	54.5
SOKOL 1513	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good	Good	8	<2	Pass	72.7
SOKOL 1514	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good	Good	4	<2	Fail	58.4
SOKOL 1515	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good	Good	6	<2	Pass	79.4
SOKOL 1516	Gloss Loss-5 rubs; Thk Loss-40 rubs	9H	Good	Good	8	<2	Pass	58.3
3M 631								
DYMAX 984 TC	Good	9H	Good	Good	4	0	Adhesion Good, Stress marks	42.8

# Aluminum, Conversion Coated + Mill-P-23377

<u>UV COATING</u>	<u>MEK RESISTANCE</u>	<u>PENCIL HARDNESS</u>	<u>DRY ADHESION</u>	<u>WET ADHESION</u>	<u>IMPACT, FORWARD, FT-LBS</u>	<u>IMPACT, REVERSE, FT- LBS</u>	<u>CONICAL MANDREL</u>	<u>GLOSS</u>
SOKOL 1510	Gloss Loss-5 rubs: Thk Loss-40 rubs	9H	Good @ UV/primer Fail @ primer/ Al	Fail	14	2	Pass	46.8
SOKOL 1511	Gloss Loss-5 rubs: Thk Loss-40 rubs	9H	Good @ UV/primer Fail @ primer/ Al	Fail	14-16	4	Pass	60.8
SOKOL 1513	Gloss Loss-5 rubs: Thk Loss-40 rubs	9H	Good @ UV/primer Fail @ primer/ Al	Fail	12	2	Pass	74.2
SOKOL 1514	Gloss Loss-5 rubs: Thk Loss-40 rubs	9H	Good @ UV/primer Fail @ primer/ Al	Fail	4	<2	Pass	61
SOKOL 1515	Gloss Loss-5 rubs: Thk Loss-40 rubs	9H	Good @ UV/primer Fail @ primer/ Al	Fail	16	16	Pass	78.5
SOKOL 1516	Gloss Loss-5 rubs: Thk Loss-40 rubs	9H	Good @ UV/primer Fail @ primer/ Al	Fail	10	2	Pass	59.2
3M 631							Adhesion Good, Stress marks	
DYMAX 984 TC	Good	9H	Good	Good	6	0	Adhesion Good, Stress marks	46.3